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[MTD 9: Lead, Zinc, Cadmium, Tin, Antimony and their
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Indian Standard
**METHODS OF CHEMICAL
ANALYSIS OF ANTIMONY**
(Revised)

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NEW DELHI 110002

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ANTIMONY

(Revised)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ANTIMONY

(Revised)

0. FOREWORD

0.1 This Indian Standard (Revised) was adopted by the Indian Standards Institution on 4 June 1965, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 This standard was first published in 1956 by the Basic Non-Ferrous Metals Sectional Committee, EDC 4. In IS:211-1958*, most of the elements specified were not covered by the methods prescribed in IS:1047-1956. A need was, therefore, felt to revise completely IS:1047-1956 so that the revision covers all the elements in the composition range prescribed in IS:211-1958*.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

1962 Book of ASTM Methods for chemical analysis of metals.
American Society for Testing and Materials.

GOST 1367 - 1953 Methods of chemical analysis of antimony. Russian Standards Body.

FURMAN (H N) (Ed) and SCOTT (W W). Standard methods of chemical analysis, 1962. Ed 5. D. Van Nostrand Company Inc., New York.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard prescribes the methods for determination of lead, copper, bismuth, iron, nickel, arsenic, tin, silver and sulphur.

NOTE — Antimony shall be determined by difference.

*Specification for antimony.

†Rules for rounding off numerical values (revised).

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with the procedure laid down in IS : 1817-1961*.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF LEAD

4.1 **Outline of the Method** — After the removal of antimony as bromide, lead is precipitated as sulphate and ignited. For low lead content the sulphate is extracted with ammonium acetate and determined colorimetrically as sulphide.

4.2 Reagents

4.2.1 *Hydrobromic Acid-Bromine Mixture* — Add 25 ml of bromine to 60 ml of hydrobromic acid (48 percent).

4.2.2 *Concentrated Hydrochloric Acid* — sp gr 1.16 (conforming to IS : 265-1950‡).

4.2.3 *Perchloric Acid* — 70 percent.

4.2.4 *Hydrobromic Acid* — 48 percent.

4.2.5 *Dilute Sulphuric Acid* — 1 : 1 (v/v).

4.2.6 *Lead Acid Solution* — Dissolve 0.5 g of lead nitrate in 200 ml of water and add with stirring 5 ml of concentrated sulphuric acid (sp gr 1.84) to the solution. Allow to stand for 24 hours and siphon or decant through a fine filter paper. Discard the precipitate.

4.2.7 *Ammonium Acetate Solution* — Dissolve 340 g of ammonium acetate in 500 ml of water. Add 20 ml of acetic acid and dilute to one litre with water.

4.2.8 *Dilute Nitric Acid* — 1 : 1 (v/v).

4.2.9 *Sucrose Solution* — Dissolve 50 g of sucrose in 50 ml of water.

4.2.10 *Hydrogen Sulphide Solution* — Saturate dilute sulphuric acid (1 : 99) with hydrogen sulphide gas.

*Methods of sampling non-ferrous metals for chemical analysis.

†Specification for water, distilled quality (revised).

‡Specification for hydrochloric acid.

4.2.11 Standard Lead Solution (1 ml = 0.0001 g of Pb) — Dissolve 0.1831 g of lead acetate $[\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2, 3\text{H}_2\text{O}]$ in water containing a little acetic acid and dilute to one litre.

4.3 Procedure (Gravimetric Method) (For Lead Content Above 0.06 Percent)

4.3.1 Take about 10 g of the accurately weighed sample in a 400-ml wide mouth flask. Add 30 ml of hydrobromic acid-bromine mixture and 30 ml of concentrated hydrochloric acid, cover and heat gently until the dissolution is complete, avoiding excessive loss of bromine.

4.3.2 Add 5 ml of perchloric acid and heat in a well-ventilated hood while swirling over an open flame until white fumes appear. Then heat moderately and intermittently to decompose lead bromide and to expel all the hydrobromic acid avoiding loss of perchloric acid. If the solution is cloudy, showing incomplete removal of antimony, add 5 ml of hydrobromic acid, and heat again to expel hydrobromic acid and decompose the bromide. Repeat the process until a fairly clear solution is obtained. Finally add 6 ml of dilute sulphuric acid and evaporate to dense white fumes to expel perchloric acid. Continue heating until the volume of solution has been reduced to about three millilitres.

4.3.3 Cool, add 100 ml of lead acid solution and boil gently to dissolve the soluble salt. Cool and allow to stand at about 50°C for one or two hours and finally bring to room temperature. Filter through a tared asbestos Gooch crucible and wash the precipitate well with lead acid solution. Wash the crucible with alcohol. Heat the precipitate first gently and then to a constant weight at dull redness (500° to 600°C) in a muffle furnace. Preserve the filtrate for the determination of copper and bismuth.

4.3.4 Carry out a blank determination following the same procedure and using the same quantity of all reagents but without the sample.

4.3.5 Calculation

$$\text{Lead, percent} = \frac{(A - B) \times 68.33}{C}$$

where

A = weight in g of lead sulphate,

B = weight in g of lead sulphate obtained in the blank, and

C = weight in g of the sample taken.

4.4 Procedure (Colorimetric Method) (For Lead Content \leq 0.06 Percent)

4.4.1 Extract the lead sulphate precipitate obtained under 4.3.3 with three 10-ml portions of hot ammonium acetate solution and wash with

hot water. Cool and transfer the solution or a suitable aliquot portion containing not more than 0.5 mg of lead to a 100-ml Nessler tube. Add water to make the solution to about 90 ml. Add 3 drops of dilute nitric acid, 1 ml of sucrose solution and 5 ml of hydrogen sulphide solution. Make up the volume to 100 ml with water, mix well and compare the colour against those of a series of standard solutions each containing measured volume of standard lead solution to which ammonium acetate, nitric acid, sucrose and hydrogen sulphide solutions have been added in the same quantities as in the test solution. It is important to add hydrogen sulphide solution simultaneously to the standard and the test solutions.

4.4.2 Calculation

$$\text{Lead, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of lead found in the sample solution, and

B = weight in g of the sample represented by the aliquot taken.

5. DETERMINATION OF COPPER AND BISMUTH BY THE PHOTOMETRIC METHOD

5.1 Outline of the Method—Copper and bismuth are precipitated as sulphides from the filtrate from lead determination (*see 4.3.3*) and dissolved in nitric acid. From an aliquot of the solution copper and bismuth are determined separately by the photometric method using ammonia and potassium iodide as complexing agents respectively.

5.2 Reagents

5.2.1 Concentrated Hydrochloric Acid—*See 4.2.2.*

5.2.2 Hydrogen Sulphide—gas.

5.2.3 Hydrogen Sulphide Wash Solution—Saturate dilute hydrochloric acid (3:97) with hydrogen sulphide gas.

5.2.4 Dilute Nitric Acid—1:1 (v/v).

5.2.5 Dilute Ammonium Hydroxide—1:1 (v/v).

5.2.6 Standard Copper Solution—(1 ml = 0.5 mg of Cu). Dissolve 0.05 g of electrolytic copper in 5 ml of concentrated nitric acid (sp-gr 1.42) and evaporate to remove excess nitric acid. Transfer the solution to a volumetric flask and dilute to 100 ml.

5.2.7 Sodium Hypophosphite Solution—30 percent (w/v).

5.2.8 Potassium Iodide Solution—10 percent (w/v).

5.2.9 Standard Bismuth Solution—(1 ml = 0.5 mg of Bi). Dissolve 0.05 g of bismuth metal in 10 ml of concentrated nitric acid, evaporate to dryness and dilute to 100 ml in a volumetric flask.

5.3 Procedure

5.3.1 Neutralize the filtrate preserved under 4.3.3 with concentrated hydrochloric acid. To this add 5 to 6 ml of concentrated hydrochloric acid and dilute the solution to about 200 ml. Warm the solution, pass hydrogen sulphide gas till precipitation is complete. Allow the precipitate to settle on a water-bath. Filter the precipitate, washing it with hydrogen sulphide wash solution. Preserve the filtrate for the determination of iron and nickel (*see 6*).

5.3.2 Dissolve the sulphides of copper and bismuth in hot dilute nitric acid containing bromine. Boil off excess of bromine and then transfer the solution to a 100-ml graduated flask. Dilute the solution to the mark. Preserve the solution for copper (*see 5.4*) and bismuth (*see 5.5*).

5.4 Determination of Copper

5.4.1 Take an aliquot of the solution preserved under 5.3.2 and neutralize the solution with dilute ammonium hydroxide and then add 5 ml in excess. Dilute the solution to 100 ml.

5.4.2 Transfer a suitable volume of the solution to an absorption cell and take the photometric reading using a light filter at 620 m μ .

5.4.3 Calibration Curve—Take a series of standard copper solutions and a reagent blank. Using the same quantities of reagents carry through all the stages as described under 5.4.1 and 5.4.2 and record the photometric readings of all the standard solutions along with the blank. Draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of copper per 100 ml of the solution.

5.4.4 Calculation—Convert the photometric readings of the sample to milligrams of copper by means of the calibration curve and calculate the percentage of copper as follows:

$$\text{Copper, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of copper found in the aliquot of the sample solution used, and

B = weight in g of the sample represented by the aliquot taken.

5.5 Determination of Bismuth

5.5.1 Transfer 10 ml or a suitable aliquot of the solution preserved under 5.3.2 to a graduated flask. Neutralize the solution with excess of

dilute ammonium hydroxide and then acidify with 2 to 3 drops of dilute nitric acid. Add 5 ml of sodium hypophosphite solution and 10 ml of potassium iodide; make the volume to 100 ml. Allow the solution to stand for 10 minutes.

5.5.2 Transfer a suitable volume of the solution to an absorption cell and take the photometric reading using a light filter at 460 m μ .

5.5.3 Calibration Curve—Take a series of standard bismuth solutions and a reagent blank. Using the same quantities of reagents, carry through all stages as described under 5.5.1 and 5.5.2 and record the photometric readings of all the standard solutions alongwith the blank. Draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of bismuth per 100 ml of the solution.

5.5.4 Calculation—Convert the photometric readings of the sample to milligrams of bismuth by means of the calibration curve and calculate the percentage of bismuth as follows:

$$\text{Bismuth, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of the bismuth found in the aliquot of the sample solution used, and

B = weight in g of the sample represented by the aliquot taken.

6. DETERMINATION OF IRON AND NICKEL BY THE PHOTOMETRIC METHOD

6.1 Outline of the Method—In the filtrate from copper and bismuth determination (see 5.3.1) iron and nickel are precipitated in ammoniacal medium as sulphides and dissolved in hydrochloric acid. Iron and nickel are then separately determined photometrically in aliquots of the solution using orthophenanthroline and dimethylglyoxime as complexing agents respectively.

6.2 Reagents

6.2.1 Hydrogen Sulphide—gas.

6.2.2 Dilute Ammonium Hydroxide—1:1 (v/v).

6.2.3 Ammoniacal Hydrogen Sulphide Wash Solution—Saturate dilute ammonium hydroxide with hydrogen sulphide gas.

6.2.4 Dilute Hydrochloric Acid—1:1 (v/v).

6.2.5 Concentrated Sulphuric Acid—sp-gr 1.84 (conforming to IS:266-1961*).

*Specification for sulphuric acid (revised).

6.2.6 Sodium Acetate-Acetic Acid Buffer Solution — Dissolve 27 g of anhydrous sodium acetate in 50 ml of water, add 24 ml of acetic acid, cool and dilute to 100 ml.

6.2.7 Hydroxylamine Hydrochloride Solution — one percent in water (*w/v*).

6.2.8 o-Phenanthroline Solution — 0.2 percent in water (*w/v*).

6.2.9 Standard Iron Solution — (1 ml = 0.02 mg of Fe). Dissolve 0.1405 g of ferrous ammonium sulphate [$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$] in 100 ml of dilute hydrochloric acid (1 : 19) and dilute to one litre in a volumetric flask. Prepare fresh as needed.

6.2.10 Perchloric Acid — 70 percent.

6.2.11 Citric Acid — 30 percent (*w/v*).

6.2.12 Bromine Water — saturated solution.

6.2.13 Dimethylglyoxime Solution — one percent solution in alcohol (*w/v*).

6.2.14 Standard Nickel Solution — (1 ml = 0.02 mg of Ni). Dissolve 0.200 g of nickel in 20 ml of dilute nitric acid with gentle heating. Cool and transfer to a one-litre volumetric flask and dilute to the mark with water and mix well. Dilute 10 ml of this solution to 100 ml in a volumetric flask with water and mix well.

6.3 Procedure

6.3.1 Boil the filtrate preserved under 5.3.1 to remove the excess of hydrogen sulphide in the solution and add an excess of dilute ammonium hydroxide. Through the warm solution, pass hydrogen sulphide gas till the sulphides of iron and nickel are precipitated. Keep the solution after passing hydrogen sulphide gas on a water-bath till all the precipitate settles down and filter off the precipitate. Wash the residue four to five times with ammoniacal hydrogen sulphide wash solution.

6.3.2 Transfer the residue to a beaker and dissolve it in dilute hydrochloric acid. Add 3 ml of concentrated sulphuric acid, heat to fumes. Cool the solution, transfer to a 100-ml graduated flask and dilute to the mark with water. Preserve the solution.

6.4 Determination of Iron

6.4.1 Transfer 10 ml or a suitable aliquot of the solution preserved under 6.3.2 to a 100-ml graduated flask. Dilute with 50 ml of water. Add 25 ml of sodium acetate-acetic acid buffer solution and shake well. Add 2 ml of hydroxylamine hydrochloride solution and 10 ml of o-phenanthroline solution, dilute to the mark and shake well.

6.4.2 Transfer a suitable portion of the solution to an absorption cell and take the photometric readings using a light filter at 490 m μ .

6.4.3 Calibration Curve—Take a series of standard iron solutions and a reagent blank. Using the same quantities of reagents carry through all stages as described under 6.4.1 and 6.4.2, and record the photometric readings of all the standard solutions along with the blank. Draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of iron per 100 ml of the solution.

6.4.4 Calculation—Convert the photometric readings of the sample to milligrams of iron by means of the calibration curve and calculate the percentage of iron as follows:

$$\text{Iron, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of iron found in the aliquot of the sample solution used, and

B = weight in g of the sample represented by the aliquot taken.

6.5 Determination of Nickel

6.5.1 Transfer 10 ml or a suitable aliquote of the solution preserved under 6.3.2 to a 100-ml graduated flask. Add 2 ml of perchloric acid and dilute the solution to 50 ml. Add 10 ml of citric acid, 5 ml of bromine water and add just sufficient dilute ammonium hydroxide to bleach the bromine colour. Add 5 ml of dilute ammonium hydroxide in excess and cool to room temperature. Add 5 ml of dimethylglyoxime solution and dilute to the mark with water and mix well.

6.5.2 Transfer a suitable portion of the solution to an absorption cell and take the photometric readings using a light filter at 530 $m\mu$.

6.5.3 Calibration Curve—Take a series of standard nickel solutions and a reagent blank. Using the same quantities of reagents carry through all stages as described under 6.5.1 and 6.5.2, and record the photometric readings of all the standard solutions along with the blank. Draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of nickel per 100 ml of the solution.

6.5.4 Calculation—Convert the photometric readings to milligrams of nickel by means of the calibration curve and calculate the percentage of nickel as follows:

$$\text{Nickel, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of nickel found in the aliquot of the sample solution used, and

B = weight in g of the sample represented by the aliquot taken.

7. DETERMINATION OF ARSENIC BY THE DISTILLATION-PHOTOMETRIC METHOD (FOR ARSENIC CONTENT LESS THAN 0.03 PERCENT)

7.1 Outline of the Method—Arsenic is distilled as arsenic trichloride in hydrochloric acid and is estimated photometrically.

7.2 Apparatus—An all-glass distillation apparatus as shown in Fig. 1 shall be used.

7.3 Reagents

7.3.1 Potassium Bisulphate—solid.

7.3.2 Concentrated Sulphuric Acid—see 6.2.5.

7.3.3 Sodium Chloride—solid.

7.3.4 Ferrous Chloride—solid.

7.3.5 Concentrated Hydrochloric Acid—see 4.2.2.

7.3.6 Dilute Nitric Acid—2 percent (v/v).

7.3.7 Carbon Dioxide—gas.

7.3.8 Hydrazine Sulphate—solid.

7.3.9 Methyl Orange Solution—0.1 percent solution in water (w/v).

7.3.10 Concentrated Ammonium Hydroxide—sp-gr 0.90.

7.3.11 Dilute Hydrochloric Acid—1:1 (v/v).

7.3.12 Standard Potassium Bromate Solution—0.01 N. Dissolve 0.2784 g of potassium bromate in water and dilute to one litre in a volumetric flask.

7.3.13 Ammonium Molybdate Solution—0.50 percent. Dissolve 0.53 g of ammonium molybdate [$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$] in water and dilute to 100 ml in a volumetric flask. Prepare fresh as needed.

7.3.14 Hydrazine Sulphate Solution—1 percent in water (w/v).

7.3.15 Standard Arsenic Solution—(1 ml = 0.01 mg of As). Dissolve 0.1320 g of arsenic trioxide in 10 ml of normal sodium hydroxide and 10 ml of 5 percent sodium carbonate solution; acidify with concentrated hydrochloric acid and dilute to one litre in a volumetric flask, transfer 100 ml of the solution to another 1-litre volumetric flask, and dilute to the mark.

7.4 Procedure

7.4.1 Take about 5 g of the accurately weighed sample in a 500-ml conical flask, add 15 g of potassium bisulphate and 20 ml of concentrated sulphuric acid. Heat the contents first at low temperature to decompose the sample and then over an open flame to expel sulphur trioxide and in

order to reduce the volume of concentrated sulphuric acid to nearly 10 ml. Cool the solution and dilute with 10 ml of water.

7.4.2 Transfer the solution to the distillation flask, with proper rinsing with minimum amount of water and set up the apparatus as shown in Fig. 1. Add 10 g of sodium chloride and one gram of ferrous chloride to the distillation flask along with 3 to 4 grains of silicon carbide.

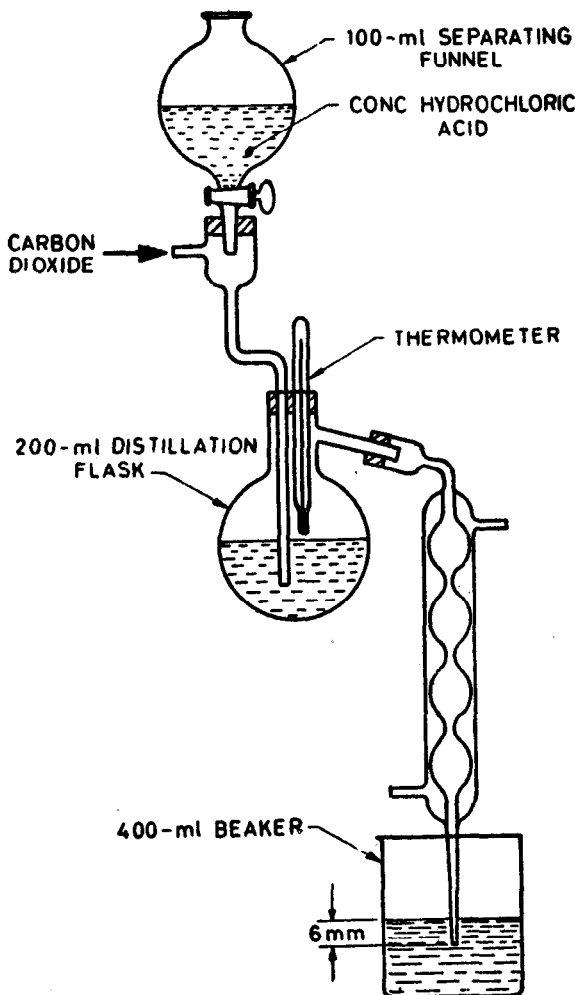


FIG. 1 DISTILLATION APPARATUS

7.4.3 Place 50 ml of concentrated hydrochloric acid in the separating funnel and place it over the distillation flask and set up the condenser of the distillation apparatus. Immerse the tip of the condenser in a 250-ml beaker containing 200 ml of dilute nitric acid. Collect the distillate for one hour. Heat the flask while dropping concentrated hydrochloric acid through the separating funnel, pass a stream of carbon dioxide into the solution at the rate of 8 to 10 bubbles per minute and collect the distillate. Heat the flask so that the temperature is maintained at 105°C.

7.4.4 To the distillate, add 10 ml of concentrated sulphuric acid and evaporate to copious fumes of sulphur trioxide. Cool the solution, add one gram of hydrazine sulphate, wash down the sides of the beaker and evaporate to fumes of sulphur trioxide. Transfer the solution to the distillation flask again when cold. Add 10 g of sodium chloride and one gram of ferrous chloride as before and distill arsenious trichloride and collect it in water in place of dilute nitric acid. Dilute the solution to 100 ml in a graduated flask. When arsenic is above 0.03 percent, dilution to 100 ml is not necessary. In that case, proceed as described under 8.

7.4.5 Take 10 ml or a suitable aliquot of the distillate solution in a 100-ml graduated flask. Add to the solution one drop of methyl orange solution and neutralize just to the yellow colour of the indicator by adding concentrated ammonium hydroxide dropwise. Add 10 ml of dilute hydrochloric acid, and one millilitre of standard potassium bromate solution. Heat the contents to discharge the methyl orange colour. Cool the solution. Heat the solution in a water-bath. Add 5 ml of ammonium molybdate solution and 2 ml of hydrazine sulphate solution. Allow the solution to remain in the water-bath for about 10 minutes for the complete development of the blue colour. Remove the flask, cool to room temperature, dilute to the mark and mix.

7.4.6 Transfer a suitable portion of the reference solution to an absorption cell and take photometric readings using a light filter at 650 m μ .

7.4.7 Calibration Curve—Take a series of standard solutions of arsenic and a reagent blank. Develop the colour by following the procedure as described under 7.4.5 and 7.4.6 and record the photometric readings of all the standard solutions along with the blank. Draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of arsenic per 100 ml of the solution.

7.4.8 Calculation—Convert the photometric readings of the sample to milligrams of arsenic by means of the calibration curve and calculate the percentage of arsenic as follows:

$$\text{Arsenic, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of arsenic found in the aliquot of the sample solution used, and

B = weight in g of the sample represented by the aliquot taken.

8. DETERMINATION OF ARSENIC BY THE IODIMETRIC METHOD (FOR ARSENIC CONTENT MORE THAN 0.03 PERCENT)

8.1 Outline of the Method—Arsenic is distilled as arsenic trichloride in hydrochloric acid and is estimated volumetrically by titration with standard iodine solution using starch solution as indicator.

8.2 Reagents—In addition to the reagents given in 7.3.1 to 7.3.11, the following reagents are required.

8.2.1 Sodium Hydroxide Solution—20 percent (w/v).

8.2.2 Methyl Red Indicator Solution—Dissolve 0.15 g of methyl red in 100 ml of water.

8.2.3 Dilute Hydrochloric Acid—1 : 1 (v/v).

8.2.4 Sodium Bicarbonate—solid.

8.2.5 Starch Solution—Make a suspension of one gram of soluble starch in about 5 ml of water and add it carefully to 100 ml of boiling water. Cool the solution before use. Prepare fresh as needed.

8.2.6 Standard Iodine Solution—0.02 N. Dissolve 2.54 g of recrystallized iodine and 8 g of potassium iodide in water. Dilute the solution to one litre with water. Store the solution in an amber-coloured glass-stoppered bottle. Standardize the solution with standard arsenious acid solution.

8.3 Procedure

8.3.1 Neutralize the distillate obtained under 7.4.4 with sodium hydroxide using methyl red indicator and then add dilute hydrochloric acid dropwise till the solution is just acidic. Cool, add about 8 to 10 g of sodium bicarbonate and 5 ml of starch solution. Titrate the solution with standard iodine solution till persistent blue colour is obtained.

8.3.2 Carry out a blank determination following the same procedure and using the same amounts of all reagents but without the sample.

8.4 Calculation

$$\text{Arsenic, percent} = \frac{(A - B) C \times 3.75}{D}$$

where

A = volume in ml of standard iodine solution required for the sample,

B = volume in ml of standard iodine solution required for the blank,

C = normality of the standard iodine solution, and

D = weight in g of the sample taken.

9. DETERMINATION OF TIN BY THE IODIMETRIC METHOD

9.1 Outline of the Method—The sample, after decomposition with acid is treated with iron powder. The filtrate is then titrated with standard iodine solution.

9.2 Apparatus

9.2.1 A 500-ml conical flask fitted with a three-hole rubber stopper containing an inlet tube for carbon dioxide, an air condenser, and a hole for a burette. This hole shall be kept closed by means of a glass rod until the titration is started. During reduction, a very slow stream of carbon dioxide shall be passed through the flask. When the reduction is complete, the flow shall be increased to maintain a protective cover of carbon dioxide during cooling and titration (see Fig. 2).

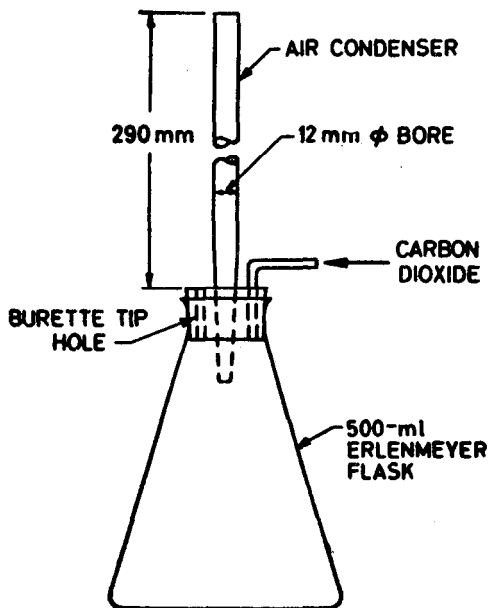


FIG. 2 APPARATUS FOR REDUCTION OF TIN

9.3 Reagents

9.3.1 Concentrated Sulphuric Acid—see 6.2.5.

9.3.2 Potassium Bisulphate—solid.

9.3.3 Concentrated Hydrochloric Acid—see 4.2.2.

9.3.4 Iron—powder.

9.3.5 Dilute Hydrochloric Acid—1:99 (v/v).

9.3.6 Carbon Dioxide Gas—oxygen-free.

9.3.7 Starch Solution—one percent.

9.3.8 Standard Iodine Solution—0.005 N. Dissolve 0.635 g of recrystallized iodine and 2 g of potassium iodide in 25 ml of water. When the solution is complete, dilute it to one litre. Preserve in a dark coloured bottle. Standardize the solution against pure tin, following the same method as described under 9.4.

9.4 Procedure

9.4.1 Take 10 g of the sample powder in a 500-ml flask. Add 100 ml of concentrated sulphuric acid and 20 g of potassium bisulphate. Digest the powder for 2 to 3 hours. Fume it strongly when the decomposition is complete. Cool the solution, add 100 ml of water and 50 ml of concentrated hydrochloric acid.

9.4.2 Add 10 g of iron powder to the solution. Warm the solution for one hour till antimony precipitates out and settles. Cool the solution and filter; wash with one percent dilute hydrochloric acid. Reserve the filtrate.

9.4.3 Dissolve the residue in about 50 ml of concentrated hydrochloric acid along with small amount of potassium chlorate, warm the solution till decomposition is complete. Boil off excess chlorine and dilute the solution to 100 ml and add 10 g of iron powder again. Filter the precipitate and collect the filtrate.

9.4.4 Combine the filtrates from 9.4.2 and 9.4.3 and transfer to a 500-ml Erlenmeyer flask. Add 5 g of iron again. Fit up the apparatus as shown in Fig. 2. Heat the solution to boiling with continuous evolution of carbon dioxide gas for about 30 minutes. After the reduction is complete, cool the contents of the flask to 10°C maintaining the atmosphere of carbon dioxide. Add a few millilitres of starch solution and titrate with standard iodine solution.

9.4.5 Carry out a blank determination following the same procedure and using the same amounts of all the reagents, but without the sample.

9.5 Calculation

$$\text{Tin, percent} = \frac{(A - B) C \times 5.935}{D}$$

where

A = volume in ml of the standard iodine solution required to titrate the sample,

B = volume in ml of the standard iodine solution required to titrate the blank,

C = normality of the standard iodine solution, and

D = weight in g of the sample taken.

10. DETERMINATION OF SILVER BY THE TURBIDIMETRIC METHOD

10.1 Outline of the Method—Silver is precipitated as sulphide from the filtrate from lead determination. The precipitate is dissolved in nitric acid and the opalescence produced by hydrochloric acid is compared with that produced under the same conditions with standard silver solution.

10.2 Reagents

10.2.1 Hydrogen Sulphide—gas.

10.2.2 Hydrogen Sulphide Wash Solution—Saturate dilute sulphuric acid solution [1:99 (v/v)], with hydrogen sulphide gas.

10.2.3 Standard Silver Solution—(1 ml = 0.001 g of Ag). Dissolve 1.0000 g of silver in 10 ml of dilute nitric acid [1:1 (v/v)] and heat until brown fumes are removed. Dilute to one litre with water in a volumetric flask.

10.2.4 Dilute Nitric Acid—1:3 (v/v).

10.2.5 Dilute Hydrochloric Acid—1:99 (v/v).

10.3 Procedure

10.3.1 Take about 10 g of an accurately weighed sample and carry out the procedure as given in 4.3.1 and 4.3.3. Dilute the filtrate to 200 ml and treat with hydrogen sulphide to precipitate heavy metals. When the precipitation is complete, filter on a medium paper, wash thoroughly with hydrogen sulphide wash solution. Wash the solution and discard the filtrate. Tear off and discard the portion of the filter paper that contains no sulphide precipitate.

10.3.2 Transfer the remainder of the paper with the precipitate to a 100-ml beaker, add 20 ml of dilute nitric acid, macerate the paper, and heat to boiling to decompose the sulphides. Cool, dilute to 75 ml with water and filter through a medium-texture paper in a 100-ml Nessler tube.

Add one millilitre of dilute hydrochloric acid. Make up the volume to 100 ml with water, mix well, wait for 5 minutes. Compare the turbidity against those of a series of standards each containing measured volumes of standard silver solution to which nitric acid and hydrochloric acid solutions have been added in the same quantities as in the test solution. It is important to add hydrochloric acid solution simultaneously to the standard and the test solutions.

10.4 Calculation

$$\text{Silver, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of silver found in the sample solution, and

B = weight in g of the sample represented by the aliquot taken.

11. DETERMINATION OF SULPHUR BY THE DIRECT COMBUSTION METHOD

11.1 Outline of the Method—Sulphur is burnt in a stream of oxygen; sulphur dioxide obtained is absorbed in standard iodine solution, the excess of which is back-titrated.

11.2 Apparatus—Assembly of apparatus as shown in Fig. 3 having the following components shall be used.

11.2.1 Oxygen Supply—Oxygen cylinder (A) containing 99.5 percent pure oxygen and provided with reduction valve for regulating the rate of oxygen.

11.2.2 Oxygen Purification Train—Consisting of flask (B) filled with potassium permanganate solution (4 percent) in potassium hydroxide solution (40 percent) and bulb (C) filled with soda asbestos in the lower part with a thin layer of glass wool and anhydron at the top.

11.2.3 Furnace—Horizontal electric tube furnace (D) with a heating capacity of 1 000°C.

11.2.4 Combustion Tube (E)—Combustion tube shall be of refractory material having an internal diameter of 15 to 20 mm and length of about 500 to 600 mm.

11.2.5 Porcelain Boat (F)—The refractory boat shall be 70 to 120 mm long, 17 to 20 mm wide and 5 to 10 mm high. The boat shall be kept to withstand a temperature of 1 000°C in an atmosphere of oxygen for a few minutes.

11.2.6 Dust Catcher (G)—filled with glass wool.

11.2.7 Absorption Flask (H)—containing starch solution.

11.2.8 Burette (J)—25-ml capacity.

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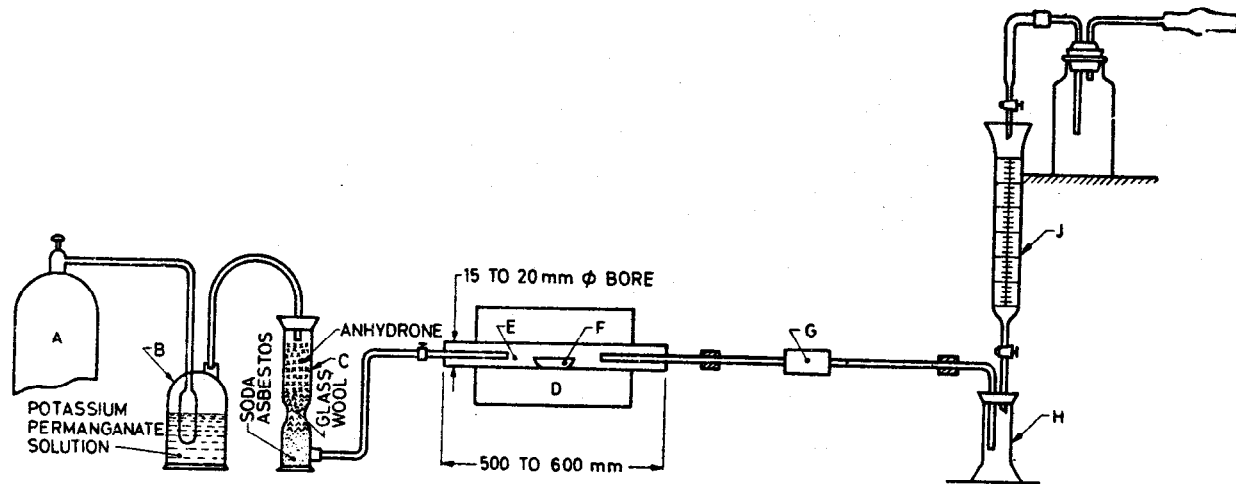


FIG. 3 APPARATUS FOR DETERMINATION OF SULPHUR BY DIRECT COMBUSTION METHOD

11.3 Reagents

11.3.1 Starch Solution — Make a suspension of one gram of soluble starch in about 10 ml of water and add it carefully to 100 ml of boiling water. Cool, add 5 g of potassium iodide and stir until potassium iodide is dissolved. Prepare the solution fresh as needed.

11.3.2 Standard Iodine Solution — 0.01 N. Dissolve 1.270 g of metallic iodine in 50 ml of potassium iodide solution (5 percent) and dilute to one litre.

11.3.3 Standard Sodium Thiosulphate Solution — 0.01 N. Dissolve 2.48 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in one litre of freshly boiled and cooled water in a sterile glass bottle. If sulphur precipitates during preparation or upon subsequent use, discard the solution and prepare a new one.

11.4 Procedure

11.4.1 Place 0.5 g of the finely ground and accurately weighed sample in the porcelain boat and introduce it in the furnace, heated before hand to 900°C . After the introduction of the sample in the combustion tube (E), close the latter immediately with a cork. Pass the stream of oxygen and burn the sample. Maintain a constant flow of oxygen through the furnace.

11.4.2 Pass the products of combustion to the absorption flask (H) containing starch solution. At the time of absorption, add a measured excess of standard iodine solution from the burette (J) and back-titrate the excess iodine with standard sodium thiosulphate solution.

11.4.3 Carry out a blank determination on the same quantity of standard iodine solution used in the test and titrate with standard sodium thiosulphate solution.

11.4.4 Calculation

$$\text{Sulphur, percent} = \frac{(A - B) C \times E}{D} \times 100$$

Where

A = volume in ml of the standard thiosulphate solution used for the blank,

B = volume in ml of the standard thiosulphate used for the excess of standard iodine solution used in the test,

C = normality of the standard thiosulphate solution,

D = weight in g of the sample taken, and

E = factor determined with a sample of known sulphur content by actual standardization.

12. DETERMINATION OF SULPHUR BY THE SULPHATE METHOD (FOR SULPHUR CONTENT ABOVE 0.1 PERCENT)

12.1 Outline of the Method

12.1.1 The sample is first attacked by bromine and subsequently brought in solution by nitric and hydrochloric acid action. After removal of antimony, sulphur is precipitated by means of barium chloride and weighed as barium sulphate.

12.2 Reagents

12.2.1 *Bromine Solution*—10 percent in carbon tetrachloride.

12.2.2 *Bromine*—liquid.

12.2.3 *Concentrated Nitric Acid*—sp gr 1.42 (conforming to IS : 264-1950*).

12.2.4 *Concentrated Hydrochloric Acid*—see 4.2.2.

12.2.5 *Ingot Iron*—chips (free from sulphur).

12.2.6 *Barium Chloride Solution*—6 percent (*w/v*).

12.3 Procedure

12.3.1 Transfer about one gram of finely ground and accurately weighed sample to a beaker and treat with 10 ml of bromine solution. Add 5 ml of liquid bromine slowly and with frequent stirring, and let stand with occasional shaking for an hour. Cool the beaker in ice water, add 15 ml of concentrated nitric acid and let stand for about 30 minutes with occasional shaking. Add 15 ml of concentrated hydrochloric acid and allow to stand at room temperature for about 30 minutes. Heat slowly to drive off carbon tetrachloride. Evaporate to syrupy consistency. (Do not overheat or permit to run dry.) Add 10 ml of concentrated hydrochloric acid and again evaporate to syrupy consistency. Add 20 ml of concentrated hydrochloric acid, heat until soluble salts are in solution. Transfer to a 500-ml Erlenmeyer flask, keeping the volume below 100 ml.

NOTE—In case the sample contains 99.8 percent antimony, a known amount of potassium sulphate should be added. From the total weight of barium sulphate found the weight equivalent to potassium sulphate added should be deducted to give the weight due to sulphur dioxide.

12.3.2 Treat with solution prepared under 12.3.1 with 5 g of ingot iron chips and allow to stand for about an hour when practically all the antimony will be removed. Filter, and wash thoroughly with water. Dilute to 160 ml, and precipitate barium sulphate by adding 125 ml of barium chloride solution from a dropping pipette at the rate of about 5 ml per minute. Let stand overnight, filter through a Gooch crucible, wash moderately with cold water, dry and ignite as usual.

*Specification for nitric acid.

12.3.3 Carry out a blank determination following the same procedure and using the same quantity of all reagents, but without the sample.

12.3.4 *Calculation*

$$\text{Barium sulphate, percent} = \frac{(A - B) \times 0.1374}{C} \times 100$$

where

A = weight in g of barium sulphate obtained from **12.3.2**,

B = weight in g of the barium sulphate obtained from the blank, and

C = weight in g of the sample taken.

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